



Combined particle mass spectrometer – Quartz crystal microbalance apparatus for *in situ* nanoparticle monitoring during flame assisted synthesis



Alexey Fomin^a, Marina Poliak^a, Igor Rahinov^b, Vladimir Tsionsky^a, Sergey Cheskis^{a,*}

^a School of Chemistry, Tel Aviv University, Tel Aviv 69978, Israel

^b Department of Natural Sciences, The Open University of Israel, Raanana 43107, Israel

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ABSTRACT

A newly developed apparatus for simultaneous *in situ* monitoring of size, mass and number concentration of a flame synthesised particle aerosol is presented. The diagnostics methodology is relying on combination of particle mass spectrometry and quartz crystal microbalance techniques. The analysis protocol includes molecular beam sampling, sorting the charged particles constituent according to mass/charge ratio (m/ze) via electrostatic deflection, and detection of the neutral particles by monitoring the variation of the quartz crystal oscillation frequency upon exposure to the particle – laden molecular beam. From these measurements, the total mass concentration and probability density distribution of m/ze could be directly determined, allowing to deduce the particle number concentration in the aerosol, at least in relative units. The feasibility of the method was demonstrated on the example of iron oxide nanoparticles (NPs) forming in $\text{CH}_4/\text{O}_2/\text{N}_2$ dual stage flame doped with iron pentacarbonyl, $\text{Fe}(\text{CO})_5$ as a precursor of iron oxide NPs. Sensitivities of 6×10^4 NP/s and 8×10^6 NP/s were achieved for charged and neutral particles, respectively. Possible implications of the obtained results on the elucidation of iron oxide NP formation mechanism are discussed.

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1. Introduction

Synthesis of metal oxide nanoparticles (NP) in flames offers several important advantages, including control over particle size and morphology and a route to large-scale production of nanomaterials [1]. Hence, it is not surprising that gas phase combustion is routinely used for production of variety of nanosculpted metal oxides. The metal oxide nanomaterials are used as pigments, opacities, flowing aids, for optical fibers, telecommunication and as highly efficient heterogeneous catalysts. Among various metal oxides, iron oxide NPs monitored in this work are used in diverse applications, including optical magnetic recording, catalysis, gas sensors, targeted drug delivery, magnetic resonance imaging, and hyperthermic malignant cell therapy (see for instance review by Lu et al. [2] and references therein). The functionalities of iron oxide NPs in various applications can be highly dependent on their characteristics, such as size, morphology and oxidation state. These characteristics depend on flame conditions (e.g., fuel/oxidizer ratio, temperature), choice of a flame fuel, particle precursor, and the position where the particles are collected [3–8]. Hence, using a flame

source allowing for a wide range of operating conditions – is of paramount importance for acquiring the ability to tune the properties of synthesized NPs. The flame source used in this work is a hybrid McKenna burner, where the primary flame supported on the porous sinter provides the energy necessary to support the central flame containing the NP precursor (see Section 2), even when the later has fuel/oxygen ratio which otherwise would not allow stable burning conditions.

Current understanding of the mechanisms governing flame assisted NP synthesis is insufficient to enable full control over particle production. One of the main challenges is the development of methodologies for *in situ* NP monitoring in reactive flows (e.g., flame), since the *ex situ* methods (electron microscopy, X-ray scattering, etc.) are very time consuming and cannot provide in a timely manner sufficient experimental data for experiments with strongly and rapidly changing flame conditions.

Particle Mobility Spectrometer (see for example [9–11]) is very effective for measurement of the particle sizes when their shape is known. Its application for low pressure flames would require further pressure reduction, which can make electrical mobility measurements under viscous gas flow regime more difficult.

A particle mass spectrometer (PMS), employing molecular beam sampling [3,12], was successively applied for *in situ* measurements of the mass distribution of charged NPs forming in flame. Measure-

* Corresponding author.

E-mail addresses: cheskis@post.tau.ac.il, scheskis@me.com (S. Cheskis).

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